

Basic Principles of Sampling and Testing

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1. INTRODUCTION

When dealing with bulk materials, the properties of a certain quantity of material, such as the contents of a preblending bed, a shift's cement production or a coal delivery, cannot be determined on the entire quantity, but have to be estimated on the basis of measurements carried out on a limited number of individual samples. The reliability of such an estimate depends on the heterogeneity of the material and on the procedure of sampling, sample preparation and analysis. A high reliability - as may be required for production or market purposes - is usually associated with comprehensive sampling, with precise preparation procedures and analytical instruments, and may thus incur considerable costs. The objective of a well designed testing procedure is, therefore, to extract the necessary information on quality parameters of a material at the lowest possible costs.

This paper reviews various aspects relevant to the design of an appropriate sampling regime, and summarizes the main factors influencing the reliability of test results. Emphasis is given to practical aspects rather than to the theory and statistics behind them; for the latter, reference should be made to the paper "Statistics" contained in the seminar documentation.

2. SAMPLING REGIME

2.1 Basic considerations

In order to decide on an adequate sampling regime and corresponding equipment, some considerations are necessary concerning the objectives of sampling, the representativity of samples from bulk materials and the material characteristics to be determined.

2.1.1 Objectives

Bulk materials can be sampled either for a mere determination of the characteristics of a certain basic material quantity or for the purpose of process control.

a) Characterization of a given basic material quantity

Examples of such materials are:

- ◆ Area of quarry to be blasted
- ◆ Materials delivered to the plant (coal, slag, gypsum)
- ◆ Kiln feed
- ◆ Dispatched cement

The sampling regime has to be adapted to the given variability of the material and to the required precision of the measurement. The complexity of sampling can vary from occasional manual grab samples to sampling regimes prescribed in norms (bulk cement) or agreed upon between seller and purchaser.

b) Control of process

Examples:

- ◆ Preparation of preblending bed
- ◆ Raw and cement mill control
- ◆ Clinker control

The sampling regime, in particular the sampling frequency, has to be adapted to the target variability of the output material, to the variability of the input materials and to the dynamics of the process, i.e. how fast and how precise corrections can be made and how fast they can be observed (dead time). This type of sampling can readily be automated, which permits a comfortable precision to be attained.

2.1.2 Representativity of samples

In order to obtain meaningful information from a sample on the characteristics of the basic material quantity, the sample must exhibit these characteristics in a representative way. Representative means that the value for a certain characteristic determined on the sample differs only randomly - and not systematically - from the corresponding mean value of the basic material quantity. For representative sampling, it is normally a prerequisite that all volume sectors of the material have an equal chance of being sampled, and that all particle sizes are correctly represented.

Representative sampling of bulk material is often rendered difficult, especially when stationary material is concerned; some of the reasons are:

- ◆ Large volumes can be inaccessible when sampling materials on stockpiles, in silos, on ships or on trucks
- ◆ Stationary materials can be stratified due to sequential deposition of different materials
- ◆ Materials with a broad particle size range tend to segregate, i.e. fines concentrate on the bottom of a material heap. If coarse and fine particles differ in characteristics of concern (e.g. composition), considerable inhomogeneities will occur, which can be an important source of sampling error. Excessive moisture behaves in a similar way.
- ◆ Separation effects also occur when materials with a broad particle size range are thrown off a quarry face or drop from one conveyor belt onto a second one running in perpendicular direction. In the latter case, strongly inhomogeneous material streams result.
- ◆ A similar effect occurs when two material streams combine on a conveyor belt, e.g. returned filter dust added to the finish cement conveyor belt.

Sampling from stationary materials should be avoided whenever possible, and replaced by sampling during loading or discharge over conveyor systems. If this cannot be done, preference should be given to traverse sampling, i.e. a primary sample should embrace the whole cross section.

Examples for traverse sampling are:

- ◆ Blast hole sampling in the quarry
- ◆ Auger sampling of a railway car

Sampling from large stockpiles can be done according to the recommendations given in norms (e.g. ASTM D 75-82 for sampling aggregates) or according to special agreements.

For the reasons mentioned above, due attention must be paid to representative sampling of conveyed materials, independent whether manual or automatic equipment is used.

2.1.3 Selection of characteristics to be measured

To keep costs to a minimum, it may be adequate to limit the number of characteristics to be determined. Preference should be given to characteristics easy to determine, which strongly correlate with other material characteristics or with process conditions.

Examples are:

Ash content of coal	correlating with	calorific value
CaO- and Fe ₂ O ₃ -content in raw-meal	correlating with	remaining elements
Literweight of clinker	correlating with	sinterzone conditions

The validity of correlation has to be periodically verified (e.g. on daily composite samples). It is evident that the chosen characteristics must not be altered during sample preparation.

2.2 Guidelines for sample size and sampling frequency

The larger the number of samples taken and the larger the extracted quantity per primary sample, the more accurate the sampling of a basic material quantity.

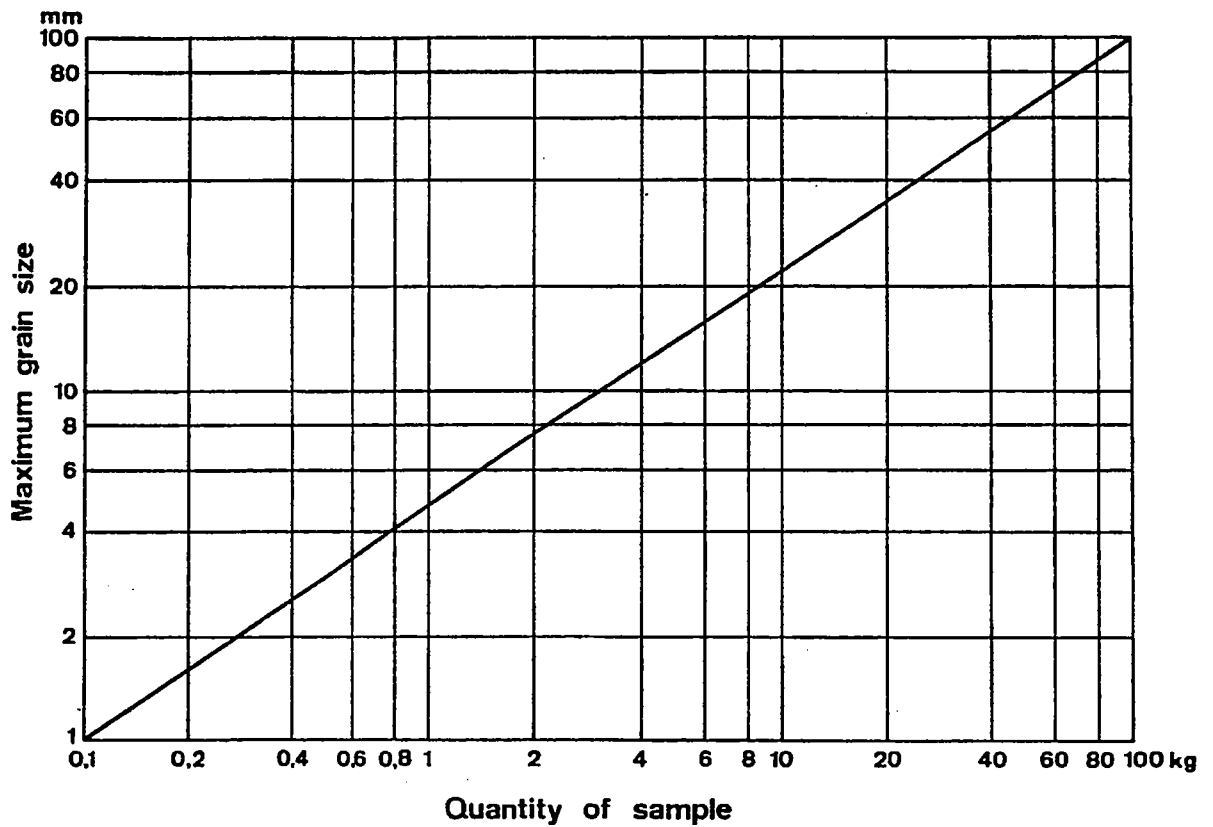
Thus, the minimum number and quantity of samples, which are required for a specific precision of the mean value of a characteristic, have to be determined. They depend upon:

- ◆ the grain size
- ◆ the heterogeneity of the material

In a first approximation, the following applies:

The minimum size of a primary sample can be related to the maximum grain size of the material. The German DIN specification states that the minimum weight of the primary sample must be at least 10 times the weight of the largest grain. This third power relationship between sample mass and maximum grain size is usually applied in linearised graphical form, as depicted in Fig. 1.

Fig. 1 Maximum grain size versus sample quantity



This graph mainly applies for coarse grained raw materials. For homogeneous fine ground materials such as raw meals or cements, the recommended sample quantity is a few hundred grams to 1 kg. For homogenous coarse grained materials, such as clinker, 1 to 3 kg are sufficient.

It may be noted that various national norm specifications relevant to the mining industry exist. The sample size / grain size relationship can differ considerably from that in Fig. 1, as illustrated by the example of manual sampling with norm scoops shown in Fig. 2.

Fig. 2: Japanese Industrial Standard M 8100

Sampling scoops and their capacities in kg for sampling bulk solids

Top Size (mm)	Scoop designation	Width (mm)	Approx. (ml)	Weight (kg) of scooped sample at various bulk densities		
				0.75	1.5	2.0
				t/m ³	t/m ³	t/m ³
5	5	50	75	0.06	0.11	0.15
10	10	75	125	0.09	0.19	0.25
15	15	100	200	0.15	0.3	0.4
20	20	100	300	0.22	0.45	0.6
30	30	150	400	0.3	0.6	0.8
40	40	150	800	0.6	1.2	1.6
50	50	260	1700	1.3	2.5	3.4

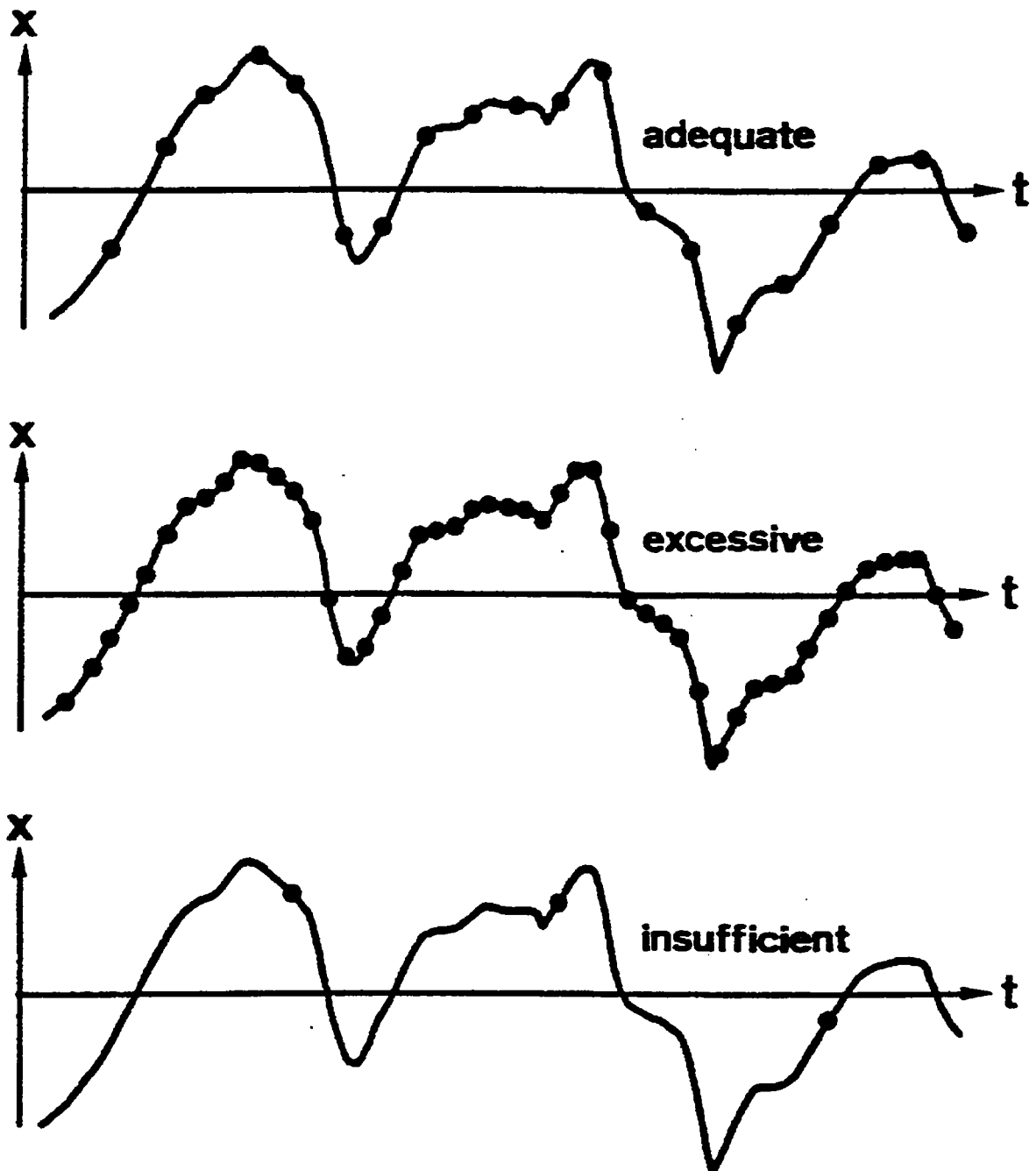
The number of primary samples - or the frequency of sampling - directly bears on the sampling precision, since the sampling variance of the mean of n-samples is n-times smaller than that of a single sample.

Hence, if the variance is known for a given basic material quantity - e.g. determined by means of a sampling experiment - the number of samples can be calculated for any desired sampling precision. This rule, however, only applies if all samples come from the same population. If the mean of the observed characteristic changes with time (trend!), samples taken at different times can no longer be regarded as originating from the same population.

Fig. 4 illustrates a reasonable sampling frequency with respect to a given materials variability, as well as too frequent and insufficient sampling.

Automatic sampling normally ensures a more than sufficient sampling frequency.

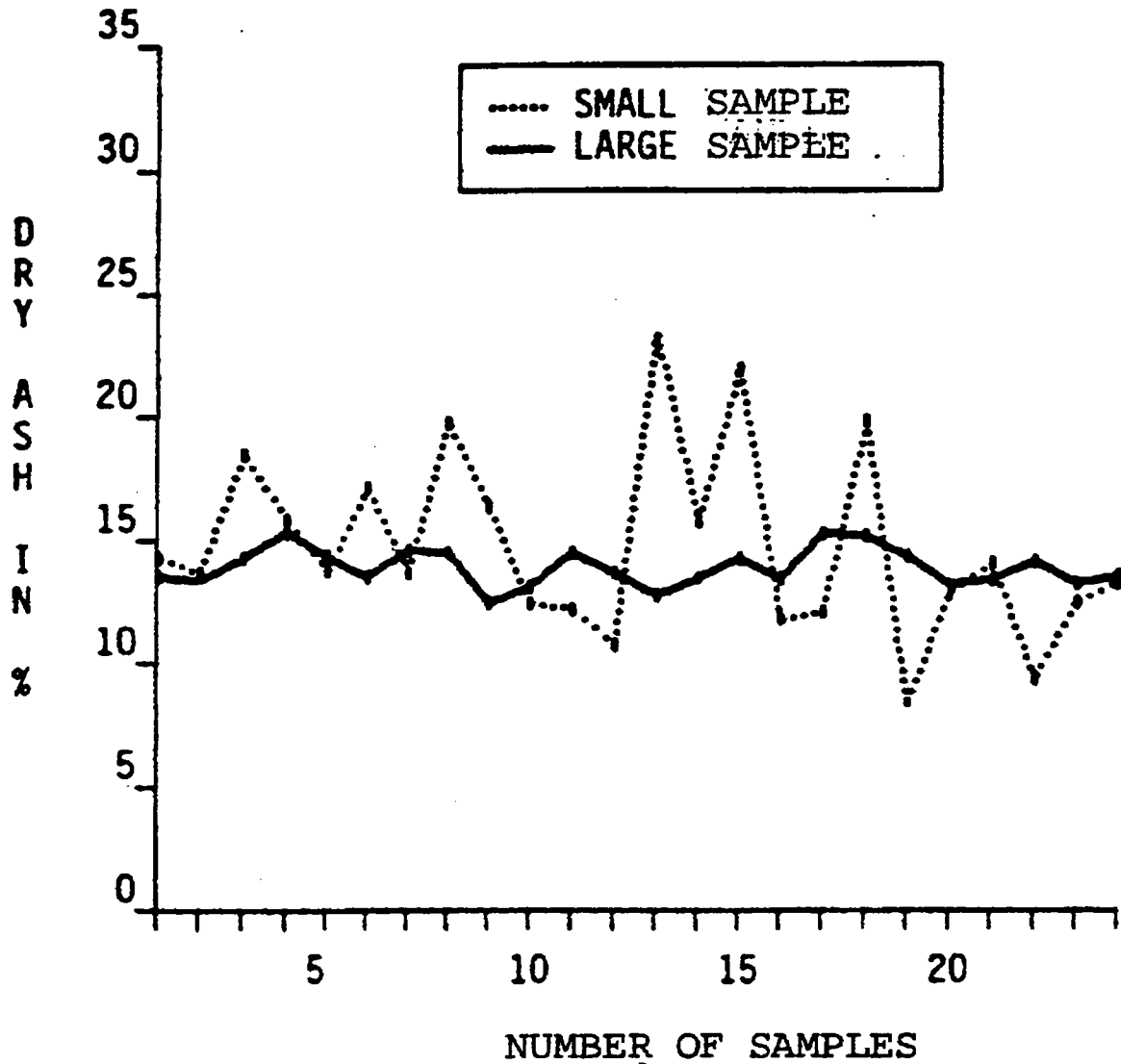
Fig. 4 Sampling Frequency



A different way to determine optimum size and number of primary samples is based on a sampling experiment in which a sufficient number of simultaneous pairs of samples of strongly different quantities are taken.

A rough evaluation of the test results - visualized in Fig. 5 - may already indicate whether much more precision is gained with a larger sample size or not. An exact evaluation can be made applying the sampling theory of Yisman [1]; an example is given in Annex 1.

Fig. 5



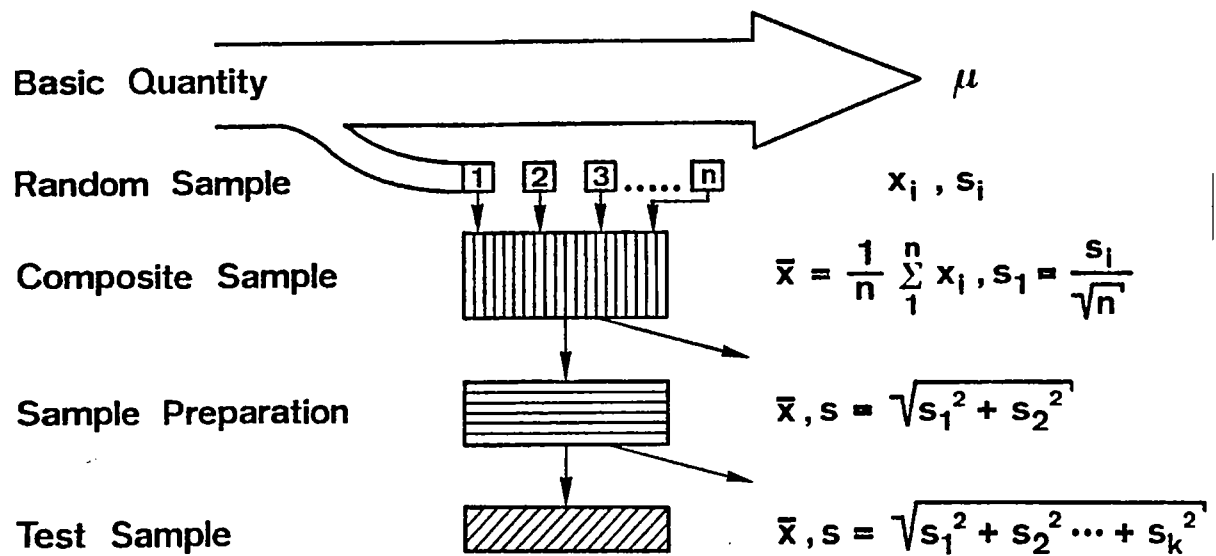
3. RELIABILITY OF TEST RESULTS

As illustrated in Fig. 6, sampling and testing procedures consist of several independent steps. The reliability of the final test result depends on the reliability of each individual step. Three types of errors can occur at each stage, namely systematic errors, random errors and gross errors. They are characterised as follows:

Systematic errors:

Systematic errors cause a bias of the estimated value for a certain characteristic with respect to the (unknown) true value. They are caused by incorrect (non-representative) sampling and sample preparation, by the malfunctioning of instruments, by inadequate calibration, or by person specific peculiarities in performing the tests.

Fig. 6



Systematic errors can be detected by cross-checking with alternative, independent testing procedures.

Examples:

- ◆ Campaign of comprehensive manual sampling to test representativity of automatic sampler
- ◆ Norm-conform CaOf-determination to cross-check conductometric determination.

Systematic errors can either be of relevance - which requires their detection and elimination -, or they can be irrelevant, usually under the condition that the error is approximately constant.

Example:

- ◆ A systematic error in determining the C_3A -content may be irrelevant when producing cement of ASTM Type I, but rather problematic when producing Type II or Type V.

Random errors:

Random errors arise due to the inherent heterogeneity of the sampled material and due to the imprecision of sampling, sample preparation and analytical techniques. The amount of random errors may be expressed by statistical measures of variability, such as variance or standard deviation (refer to paper "Statistics").

Random errors cannot be eliminated, but they can be reduced by :

Repetition, i.e. extraction of more primary samples, performance of measurements on several test samples, repeating measurements on individual test samples.

The variance (s^2) of the mean value of n measurements is n -times smaller than the variance of a single measurement

$$s^2(\bar{x}) = \frac{s^2(x)}{n}$$

Applying more precise procedures and instruments for sample preparation and analysis

Random errors of a sequence of independent testing activities add up to the total random error of the final result according to the law of error propagation (see Fig. 6).

$$s_{total} = \sqrt{s_1^2 + s_2^2 + \dots + s_n^2}$$

From this law of error propagation, the following important conclusions can be derived:

- ◆ an error which has been committed in the testing procedure cannot be compensated by increased precision in the subsequent testing steps
- ◆ the individual errors of the sequence should be as small as possible and of similar size: There is no point in increasing the precision of one step as long as others are decisive for the overall error. Therefore, it is necessary to determine the precision of the individual steps in each control.

Gross errors

Gross errors arise from the wrong performance of procedures, the wrong reading of scales, errors in copying data, miscalculations etc. Such errors can considerably influence statistical evaluations. They may show up as outliers in a series of data.

LITERATURE:

[1] Merks J.W.: Sampling and weighing of bulk solids.
Trans Tech. Publications, 1st Ed., Clausthal-Zellerfeld, Germany (printed in USA)

Additional literature:

Hasler R.:
Some aspects of process technology of raw material preparation.
HMC-report PA 74/6167/E

Gy P.:
L'échantillonnage de minerais en vrac.
Mémoires du Bureau de Recherches Géologiques et Minières No. 67,
Paris 1971

4. ANNEX 1

OPTIMUM SIZE AND NUMBER OF INCREMENTS ACCORDING TO THE THEORY OF VISMAN [1]

The total variance of a sampling and testing procedure is split up into the terms:

$$s^2(t) = \frac{s^2(c)}{am \cdot n} + \frac{s^2(d)}{n} + s^2(p) + s^2(a)$$

$$= s^2(s) + s^2(p) + s^2(a)$$

in which	$s^2(t)$	= total variance of mean value
	$s^2(s)$	= sampling variance
	$s^2(c)$	= composition variance
	$s^2(d)$	= distribution variance
	$s^2(p)$	= preparation variance
	$s^2(a)$	= analysis variance
	am	= size of primary sample (kg)
	n	= number of primary samples

The composition variance and the distribution variance can be determined by means of a sampling experiment, in which a number (~ 30) of pairs of primary samples differing strongly in size are taken and analysed.

$$s^2(c) = \frac{am(1) \cdot am(2) \cdot [s^2(1) - s^2(2)]}{am(2) - am(1)}$$

$$s^2(d) = s^2(2) - \frac{am(1) \cdot [s^2(1) - s^2(2)]}{am(2) - am(1)}$$

in which	$s^2(c)$	= composition variance
	$s^2(d)$	= distribution variance
	$s^2(1)$	= variance for set of small samples
	$s^2(2)$	= variance for set of large samples
	$am(1)$	= average mass of small samples
	$am(2)$	= average mass of large samples

The optimum size of primary samples is then given by

$$m(opt) \frac{s^2(c)}{s^2(d)}$$

and the optimum number of primary samples to obtain a required total variance:

$$n = \frac{s^2(d) + s^2(c) / m(opt)}{s^2(t) - [s^2(p) + s^2(a)]} = \frac{s^2(d) + s^2(c) / m(opt)}{s^2(s)}$$

Example (compiled from [1]):

A coal delivery is sampled by means of 24 pairs primary samples of about 100g and 50 kg size. The primary samples of small size are entirely dried and ground to analytical fineness. The primary samples of large size are dried, crushed and split and only a partial samples ground to analytical fineness. The ash content is determined in duplicate. The results are given in tables A1 and A2.

Table A1: SAMPLING EXPERIMENT WITH SMALL SAMPLES

Sample Number	Mass in g	First	Ash on Dry Second	Basis Δ	Mean
1	89	14.20	14.23	0.03	14.22
2	126	13.27	13.66	0.06	13.69
3	152	18.44	18.42	0.02	18.43
4	109	15.87	15.75	0.12	15.81
5	149	13.71	13.81	0.10	13.76
6	87	17.10	17.13	0.03	17.12
7	110	13.64	13.70	0.06	13.67
8	142	19.71	19.64	0.07	19.68
9	111	16.34	16.31	0.03	16.32
10	140	12.49	12.38	0.11	12.44
11	121	12.19	12.21	0.02	12.20
12	150	10.73	10.75	0.02	10.74
13	160	23.24	23.20	0.04	23.22
14	96	15.71	15.80	0.09	15.76
15	105	21.83	21.89	0.06	21.86
16	132	11.78	11.73	0.05	11.76
17	141	12.14	12.05	0.09	12.10
18	123	19.84	19.67	0.17	19.76
19	135	8.34	8.37	0.03	8.36
20	150	12.80	12.85	0.05	12.82
21	108	14.02	13.91	0.11	13.96
22	99	9.35	9.31	0.04	9.33
23	102	12.46	12.38	0.08	12.42
24	129	13.15	13.18	0.03	13.16
Mean	123.9			0.063	14.69
Variance				0.0031	14.1448
Standard Deviation				0.0558	3.7610
Coefficient of Variation					25.6
Variance of the mean					0.5894
Standard deviation of the mean					0.7677
Coefficient of variation of the mean					5.2%

Sampling variance : total variance minus variance of analysis

$$s^2(s) = 14.1448 - 0.0031 = 14.1417$$

Table A2: SAMPLING EXPERIMENT WITH LARGE SAMPLES

Sample Number	Mass in g	First	Ash on Dry Second	Basis Δ	Mean
1	53.0	13.61	13.34	0.27	13.48
2	53.2	13.15	13.46	0.31	13.30
3	55.9	14.34	14.20	0.14	14.27
4	41.1	15.33	15.26	0.07	15.30
5	46.1	14.17	14.51	0.34	14.34
6	40.6	13.60	13.41	0.19	13.50
7	48.8	14.76	14.38	0.38	14.57
8	50.2	14.25	14.72	0.47	14.48
9	55.7	12.54	12.29	0.25	12.40
10	48.1	13.02	13.21	0.19	13.12
11	43.4	14.33	14.74	0.41	14.54
12	48.4	13.70	13.63	0.07	13.66
13	45.3	12.80	12.68	0.12	12.74
14	40.1	13.26	13.64	0.38	13.45
15	41.8	14.09	14.41	0.32	14.25
16	48.0	13.52	13.28	0.24	13.40
17	56.2	15.44	15.11	0.33	15.28
18	50.3	15.07	15.28	0.21	15.18
19	48.0	14.40	14.26	0.14	14.33
20	44.3	13.06	13.22	0.16	13.14
21	48.5	13.22	13.47	0.25	13.34
22	41.6	14.26	13.90	0.36	14.08
23	39.9	13.18	13.12	0.06	13.15
24	52.3	13.44	13.56	0.12	13.50
Mean	47.53			0.241	13.87
Variance				0.0455	0.7903
Standard Deviation				0.2134	0.8890
Coefficient of Variation					6.6
Variance of the mean					0.0329
Standard deviation of the mean					0.1815
Coefficient of variation of the mean					1.3%

$s^2(s) = 0.7903 - 0.0455 = 0.7448$

calculation of composition and distribution variance:

$s^2(c) = \frac{123.9 \cdot 47530 \cdot (14.1417 - 0.7448)}{47530 - 124} = 1664.2$

$s^2(d) = \frac{0.7448 - 123.9 \cdot (14.1417 - 0.7448)}{47530 - 124} = 0.71$

Hence:

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$$\text{Optimal mass } m(\text{opt}) = \frac{1664.2}{0.71} = 2343\text{g} \quad \underline{2.3 \text{ kg}}$$

(entire primary sample can be prepared to analytical fineness, thus only analytical variance to be considered)

Optimal number of primary sample:

$$n = \frac{0.71 + 1664.2/2343}{s^2(t) - s^2(a)} = \frac{0.71 + 0.71}{s^2(s)} = \frac{1.42}{s^2(s)}$$

Error of mean (95% conf intev.)	Coeff. of Variation	s	s ² (t)	s ² (s) = s ² (t) - 0.0031	n
± 10%	5%	0.7	0.49	0.4869	3
± 5%	2.5%	0.35	0.12	0.1194	12
± 2%	1%	0.14	0.02	0.0165	86
± 1%	0.5%	0.07	0.0049	0.0018	(800))*

* Analytical variation outweighs sampling variation, i.e. error of mean is determined by analytical precision and not by sampling regime.

